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Low Volatile Organic Compound Containing Wash Primer for Letterkenny Army Depot

by Fred Lafferman, Daniel Pope, and John Escarsega

ARL-TR-6166 **September 2012**

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14. ABSTRACT
Currently, only wash primer conforming to military specification DOD-P-15328 is authorized to be used as a spray in place pretreatment for multi metal applications prior to the application of the chemical agent resistant coatings (CARC) systems. Pennsylvania State Environmental regulations regulate the volume of volatile organic compounds in coatings such as DOD-P-15328, Wash Primer Pretreatment. Laboratory testing was conducted with two experimental wash primers and evaluated for performance as compared to the baseline, DOD-P-15328. These experimental wash primers were formulated with lower percentages of volatile organic compounds to comply with Pennsylvania State laws. Laboratory testing indicates that these products provide equal performance to DOD-P-15328, but final approval for implementation will be based upon outdoor exposure testing at Cape Canaveral and a demonstration at Letterkenny Army Depot in Chambersburg, PA.

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CARC, corrosion, coating

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1. Introduction

Pretreatments containing hexavalent chromium have been the industry standard for chemical agent resistant coatings (CARC) systems due to exceptional adhesion, substrate passivation, and corrosion inhibition properties. For the pretreatment of ferrous and non-ferrous surfaces that are not adaptable to an immersion process, wash primer conforming to DOD-P-15328 has evolved as the standard process in the CARC coating system. Zinc chromate pigments are the primary pigments used in DOD-P-15328 wash primer, providing the major advantages of enhanced corrosion and flash rust resistance.

Military Specification DOD-P-15328 for wash primer has not been revised since 1978. Since then, the need for spray application pretreatments that can be used on mixed metals has not changed, but environmental concerns have caused an urgent need to find a viable replacement to DOD-P-15328.

Military specification DOD-P-15328 describes low solids material containing extremely high volatile organic compound (VOC) content, phosphoric acid, zinc chromate, and has hazardous air pollutants (HAPs) used as a multi-substrate pretreatment for military assets. Over the past several years, the U.S Army Research Laboratory (ARL) Coatings Technology Team has reformulated all of the CARC, including the epoxy primers and the camouflage polyurethane topcoats, to eliminate organic and inorganic HAPS and reduce the volatile organic compounds to conform to Federal and State Air Quality Standards. Many efforts have attempted to find a replacement that works for all applications currently using wash primer. These efforts have not produced a product that is viable and practical for use as a direct replacement for DOD-P-15328.

Recently, Letterkenny Army Depot in Chambersburg, PA has been receiving fines on a regular basis due to total VOC emissions with non-Pennsylvania compliant coatings. While chromate is an environmental issue, the biggest issue facing Letterkenny is the VOC emissions. Therefore, the need is for a lower VOC wash primer that may contain chromate as a short term remedy.

ARL and the U.S. Army Aviation and Missile Command is working with Sustainable Painting Operations for the Total Army to test chromate containing wash primers with lower VOC emissions compared to the current specified wash primer DOD-P-15328. With the assistance of NCP Coatings Inc. and Spectrum Coatings Inc., two different systems were developed with the same basic pigmentation and resin system as DOD-P-15328, but with different solvent packages that include Environmental Protection Agency (EPA) exempt solvents that will reduce the VOC emissions of Letterkenny Army Depot.

2. Products

Two experimental products were tested. These products were from NCP Coatings, Inc (NCP) of Niles, Michigan, with manufacturer's designation N-9025A/B, and Spectrum Coatings, Inc. (Spectrum) of Providence, RI, with manufacturer's designation GWP-EX100 A/B. The products tested were required to contain the same polyvinyl butyral resin system and zinc chromate pigment as the current DOD-P-15328 (*1*). For each product, the solvent package was changed to include EPA exempt solvents to bring the VOC of the formulations to 3.5 lb/gal or lower. The current specification formulation, when properly reduced, will have a VOC content of 6.1 lb/gal.

The experimental wash primers were tested over 2024-T3 aluminum and SAE 1008 steel. Both substrates had a set of panels with a bare mill finish and a blasted profile finish. Panels that were blasted were done so with material that is commonly used at Letterkenny during the process of preparing surfaces for refinishing.

Three different CARC epoxy primers were used to coat the wash primer. MIL-DTL-53022 (*2*) is a solvent borne epoxy primer that is lead and chromate free; NCP's type II (high solids, 3.5 lb/gal) product, coded N-1981, was used for testing. MIL-DTL-53030D (*3*) is a water reducible epoxy primer that is lead and chromate free; Deft Inc's type I (basic corrosion performance) product, coded 44-W-7, was used. MIL-PRF-23377J (*4*) is a solvent-borne high solids epoxy primer; for this test, type I (standard pigments) class N material was used. Class N contains non--chromate based corrosion inhibitors. Hentzen Coatings Inc's product, coded 16708TEP/16709CEH, and Deft's product, coded 02GN084, were used in testing. All of the epoxy primers used had been tested and received approval for the qualifying activity to be listed on the Qualified Products Database.

3. Testing

Testing requirements were established based upon specifications MIL-DTL-53022 and MIL-DTL-53030. The experimental wash primer's results would be compared to the results of the DOD-P-15328 wash primer, which is being used as the control. Testing was designed to validate the ability of the wash primer to enable the primer to pass the minimum requirement of the specification and to compare the experimental materials to the current specified wash primer.

Spraying and mixing properties were observed at Letterkenny Army Depot. Because these materials were tested as possible replacements for DOD-P-15328 at Letterkenny, it is important that the experimental products are able to be properly mixed and sprayed in the facilities at Letterkenny.

Adhesion was tested in accordance of American Society for Testing and Materials (ASTM) Standard 3359D, method B (*5*). This method required cross hatching of the coating system to the substrate and a specified tape placed firmly on the area, which had been cross hatched. After 60 s, the tape was pulled off at a near 180° angle. The requirement is for a rating of 4B or better. A rating of 4B requires that less than 5% of the coating system is removed by the tape.

The flexibility of the coating system was tested in accordance with ASTM Standard D522 (*6*). Panels were bent over a ¼ inch mandrel. Once bent, coatings must show no sign of cracking or pulling from the panel. Due to the nature of this test, blast cleaned aluminum and steel panels were not tested for flexibility as they are thicker panels and will not bend over the ¼ inch mandrel.

The coating systems were tested for water and JP8 jet fuel immersion resistance. The coated panels were exposed for seven days; one set of panels in de-ionized water and one set of panels in JP8. After the seven-day immersion, the panels are checked at 2 and 24 h intervals. After 2 h, the panels must have shown no signs of the coating system having been compromised. There must be little to no softening of the coating to be considered a passing system. After 24 h, the hardness of the coating system must be fully recovered and the adhesion must rate, as well as a panel that was not exposed in the fluids.

Corrosion resistance was tested using both ASTM Standard B117 (*7*) neutral salt fog exposure chambers and GMW14872 (*8*) cyclic corrosion chambers. All of the panels exposed for these tests received an "X" scribe prior to exposure. The salt fog panels were examined immediately after removal from the salt spray test and were to show no more than a trace of rusting (ASTM D610 (*9*), table 1, rust grade 9) or corrosion, and no more than five scattered blisters, none larger than 1 mm in diameter for unscribed regions. Scribed areas were to have ratings as specified in ASTM D1654 method A (*10*) of not less than 6 for steel or 8 for aluminum panels. The cyclic corrosion panels were evaluated using ASTM method D1654 and were to have a rating of not less than 7. There were to be no more than five scattered blisters in the unscribed areas. Table 1 reflects the specified requirement for B117 exposure length.

Table 1. B117 exposure requirement by specification and substrate.

—	**Substrate**	**B-117(h)**	**B-117 Creepage Rating**
MIL-DTL-53022 Type II	CRS	336	6
	AL 2024 T-3	NA	—
MIL-DTL-53030 Type I	CRS	336	6
	AL 2024 T-3	336	8
MIL-PRF-23377 Class N	CRS	NA	—
	AL 2024 T-3	2000	8

Panels for ASTM B117 and GMW14872 were labeled to track performance. Tables 5–20 use the codes to display the results. Each label is broken down into three sections split by a "—". Figure 1 shows the three part code. The label starts with the code for the wash primer used, followed by the primer used and concluded with the id number for that panel. The codes can be found in table 2.

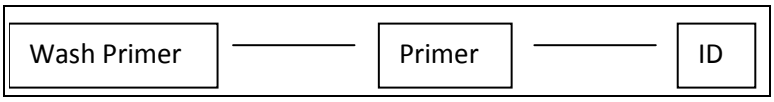

Figure 1. Format for codes in tables 5–20.

Table 2. Codes for corrosion panels.

Washprimer	Code
NCP	N
Spectrum	SP
MIL-P-15328	328
Primer	**Code**
MIL-DTL-53022	022
MIL-DTL-53030	030
MIL-PRF-23377	23377

The coating systems being tested were also topcoated with MIL-DTL-53039D (*11*) type IX for outdoor exposure. These panels will be exposed at the Kennedy Space Center, Beachside Atmospheric Test Facility. The panels will be exposed until failure. The results for the coating systems with the experimental wash primers will be compared to results from the control wash primer. Testing at this static site is expected to last up to two years.

4. Results

All of these products were sprayed at Letterkenny Army Depot during two separate trips. During the first trip, which occurred 23 March 2010, all but the blast cleaned aluminum and steel panels for outdoor exposure were coated.

The control wash primer was sprayed as it would have been applied in production at Letterkenny. While the proper mix ratio of four parts component A to one part component B mix was being used, the required thinner was not, at the direction of Letterkenny personnel. DOD-P-15328 is meant to be thinned using one part isopropyl alcohol. Without the isopropyl alcohol, the specification wash primer sprays inconsistently and cannot properly coat the substrate. Figures 2 and 3 show the result of specification wash primer not properly thinned for application, this finish is very inconsistent.

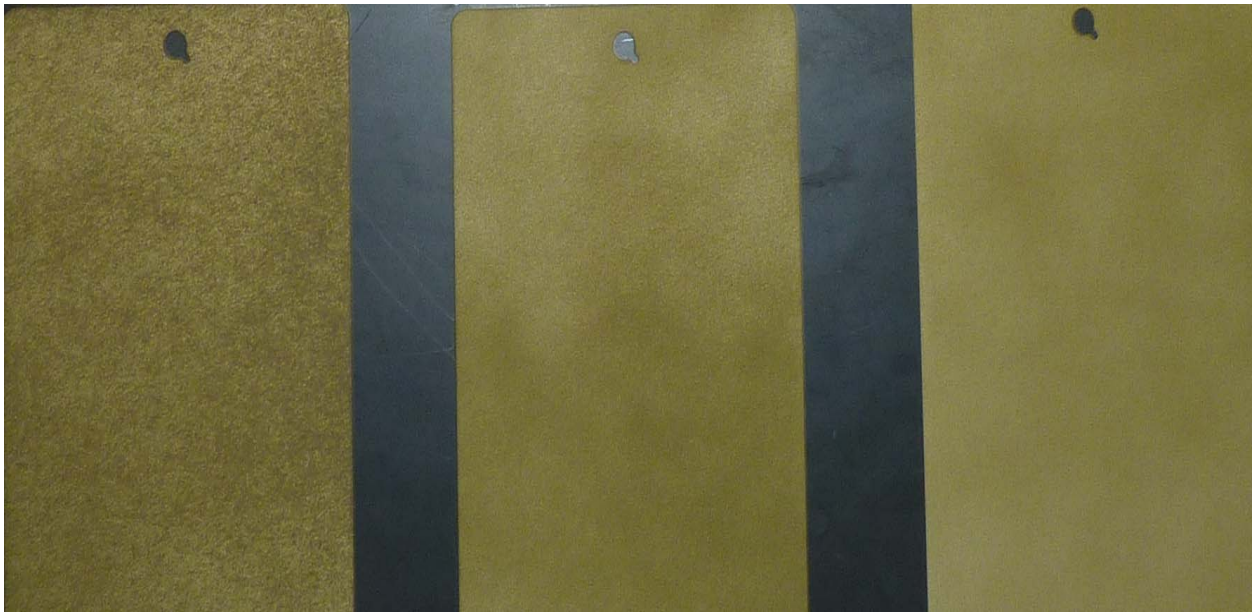

Figure 2. Left to right: DOD-P-15328, NCP experimental wash primer, Spectrum experimental wash primer. All the coating is over cold rolled steel with a bare mill finish.

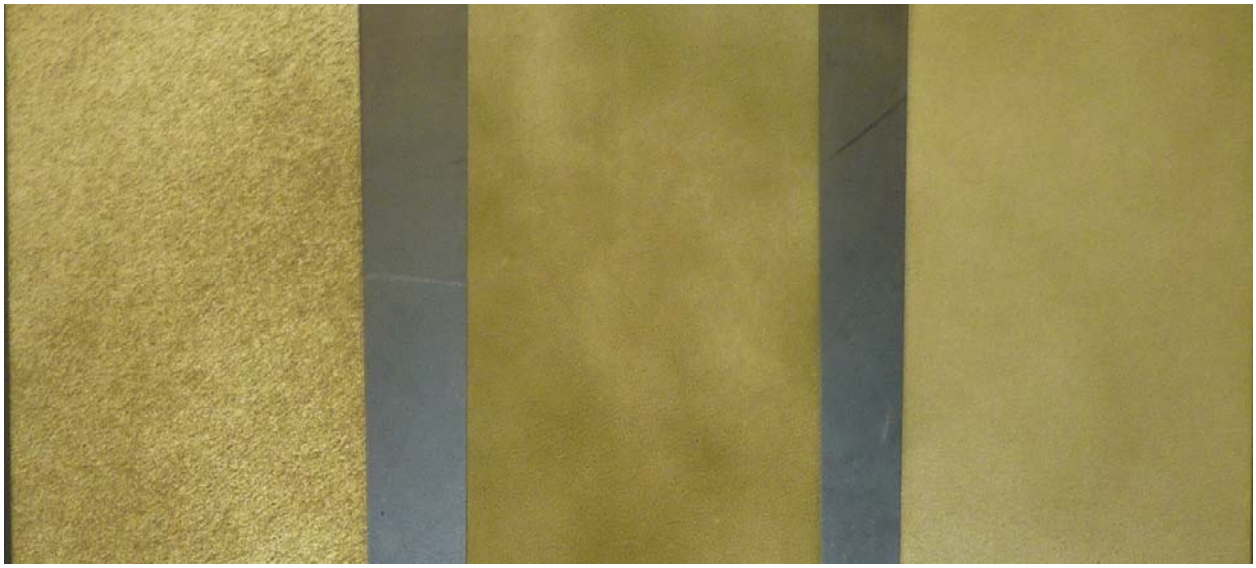

Figure 3. Left to right: DOD-P-15328, NCP experimental wash primer, Spectrum experimental wash primer. All the coating is over 2024-T3 aluminum with a bare mill finish.

During the second trip to coat panels, which occurred 19 October 2010, the control wash primer was sprayed with the proper volume of the specified thinner, isopropyl alcohol. With the thinner, the DOD-P-15328 wash primer sprayed as the specification requires and provided a smooth and consistent finish. The resulting finish was the same as the two experimental wash primers, when thinned properly.

NCP's experimental wash primer has a mix ratio of one part component A to one part component B. This product does not require any additional thinner. NCP's product was sprayed in accordance with guidance that was provided by the vendor's technical data sheet during both sessions. This product mixed together very easily and sprayed a very consistent film. These samples can be seen in the center of figures 2 and 3.

Spectrum's experimental wash primer has a mix ratio of four parts component A to one part component B and three to four parts exempt thinner. The original mixture used three parts of exempt thinner. This was found to be not enough thinner. With three parts exempt thinner, Spectrum's experimental wash primer tended to cobweb when sprayed. When the mixture was made with four parts exempt thinner, the product sprayed well. It provided a smooth and consistent finish that can be seen on the right side of figures 2 and 3.

The three different primers were sprayed on top of each set of wash primer. When MIL-DTL-53022 type II was applied, there was no issue with the primer over the wash primers. Issues were witnessed with the application of MIL-DTL-53030 type I over all of the wash primers. As the primer started to dry, it would begin to discolor. Also, development of micro blistering could be seen on many of these panels. Hentzen's MIL-PRF-23377 type I class N was used to spray the first session. Once sprayed, the primer could be seen pulling from the edges. Figure 4 demonstrates the edge pull on a blasted steel panel that had been treated with control wash primer. The corner of the panel appears to be completely void of the primer due to the edge pull. This edge pull occurred over all of the wash primers. For the second session on 19 October Deft's MIL-PRF-23377 type I class N was used. This change was made due to the fact that severe edge pull could significantly affect the results during corrosion testing. The Deft primer did not exhibit edge pull. Also observed were drying times. While MIL-DTL-53022 and MIL-DTL-53030 primers were dry to touch in under an hour, MIL-PRF-23377 primer took a longer time to set to touch. Dry to touch was observed ~4 h after the application. It was also observed that there was no effect on drying time with the experimental wash primers when compared to the control samples.

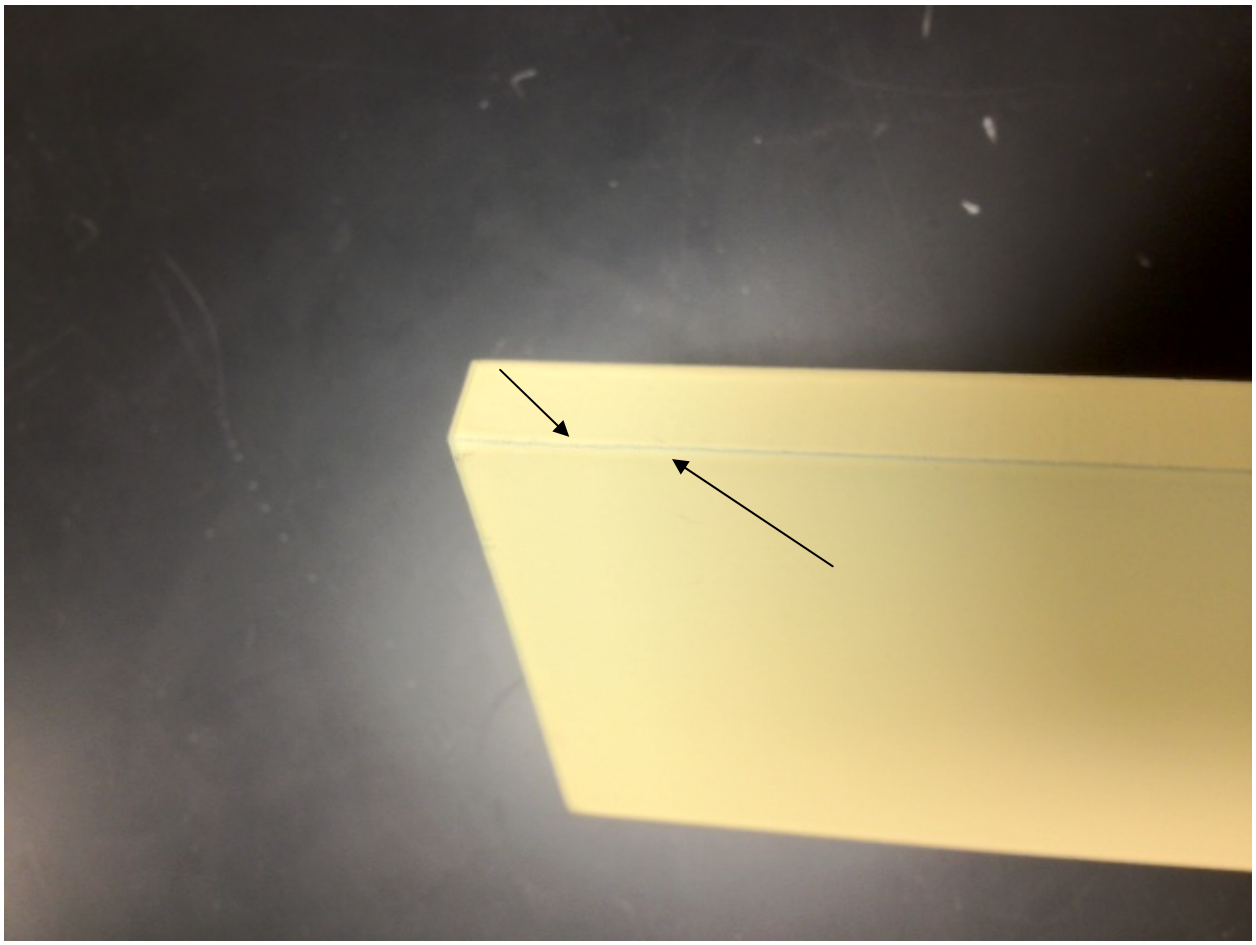

Figure 4. Edge pull with MIL-PFR-23377 class N primer over blasted steel.

The dry film build of the different samples were taken and recorded in table 3. Results for the blast cleaned panels were very difficult to obtain due to the surface profile of the test panel. Three panels of each set were tested in five spots. These were averaged and the results are displayed in table 3. The samples sprayed with MIL-PRF-23377 class N were found to be much thicker than the other primers in testing.

Regarding the samples using the MIL-PRF-23377 primer, both cold rolled steel panel types, as well as the smooth aluminum were prepared and had their dry film thickness measured during the first session of spraying. These samples used the MIL-PRF-23377 provided by Hentzen. The blasted aluminum panels that utilized the same preparations were prepared and measured during the second spraying session and used MIL-PRF-23377 provided by Deft.

Table 3. Dry film thickness.

Wash Primer/Primer System	Smooth CRS	Smooth AL	Blasted Steel	Blasted AL
MIL-DTL-53022	—	—	—	—
DOD-P-15328	2.03	2.51	3.22	3.73
NCP	2.05	2.05	4.29	3.86
Spectrum	1.74	2.2	3.85	3.68
MIL-DTL-53030	—	—	—	—
DOD-P-15328	1.26	1.29	2.53	1.88
NCP	1.6	1.01	2.41	1.56
Spectrum	1.05	1.02	2.93	2.08
MIL-PRF-23377 Class N	—	—	—	—
DOD-P-15328	3.13	2.81	4.54	5.1
NCP	3.11	2.92	5.3	5.5
Spectrum	3.3	3.64	6.4	4.91

Film thickness on all of the blasted substrates was higher than on the smooth panels. The blasted panels had a blast profile of 1–2 mils. Because of this, the total build of the wash primer and primer had to be thicker to insure that all of the peaks on the surface were adequately coated.

Cross-hatch adhesion testing showed that there was little or no difference between each of the wash primers. Both the NCP and Spectrum experimental wash primers performed, as well as the control. There were issues with the samples primed with Hentzen's MIL-PRF-23377 type I class N. The primer chipped very easily when being scratched for the test. It behaved this way over the experimental and specified wash primers. Deft's MIL-PRF-23377 type I class N was coated on the 2024-T3 aluminum panels with the blast profile. The same adhesion failures were not witnessed on the blasted aluminum panels. Table 4 details the results of the testing.

Table 4. Cross-hatch adhesion results.

AL 2024	—	—	—	AL 2024 Blasted	—	—	—
—	53022	53030	23377	—	53022	53030	23377
15328	4B	5B	3B	15328	4B	4B	5B
NCP	4B	4B	3B	NCP	4B	4B	4B
Spectrum	4B	4B	2B	Spectrum	4B	4B	5B
CRS	—	—	—	HRS Blasted	—	—	—
—	53022	53030	23377	—	53022	53030	23377
15328	5B	4B	3B	15328	4B	4B	4B
NCP	4B	4B	2B	NCP	4B	4B	3B
Spectrum	5B	4B	2B	Spectrum	5B	4B	3B

A result of 4B or 5B is considered passing for a CARC system. All of the panels coated with MIL-DTL-53022 and MIL-DTL-53030 generated passing results. The failures with the MIL-PRF-23377 products can be attributed to the film thickness. The film thickness was about 1 mil greater than specification requirements. This can probably be attributed to the fact that

MIL-PRF-23377 has an extremely low viscosity and required two coats to prevent sagging and running. Also, Letterkenny painters have had no experience with the application of this primer.

Flexibility was tested on the non-blasted substrates. There were no failures with the experimental wash primers that were coated with MIL-DTL-53022 or MIL-DTL-53030. With the Hentzen MIL-PRF-23377 type I class N, there was some cracking at the point of the bend. MIL-PRF-23377 is not normally tested for flexibility with ASTM D522. Also, film thickness was a contributing factor to the issues observed with systems primed with MIL-PRF-23377 for flexibility. The NCP and Spectrum experimental wash primers performed at the same level as the control wash primer with all primers for this test.

The immersion testing, water and JP8, reflected passing results for all of wash primers. The hardness results of all of the samples were similar to unexposed samples after two hours. When tested at 24 h, the hardness of each of the samples was fully recovered. Adhesion after 24 h was identical to what was observed for dry cross-hatch adhesion.

ASTM B117 testing results can be found in tables 5–12. Tables 5 and 6 have the results for cold rolled steel with the smooth finish. Results are fairly similar for MIL-DTL-53022 and MIL-PRF-23377 at the specification's required measurement after 336 and extended hours of 672. MIL-DTL-53030 did very poorly with all wash primers. Passing results should have been recorded at no less than 336 h, but all the samples failed before that point.

For tables 5–12 and tables 13–20, green shaded areas represent passing results in accordance with the ratings. Yellow shaded areas represent passing results that are showing signs of corrosion. Orange shaded areas represent marginal failing results. Red shaded areas represent failing results. Black shaded areas depict a complete failure and the product was pulled from testing. All testing period following a black shaded box will be gray denoting that the sample was not graded at that interval due to the pervious failure. All panels were evaluated using two different methods. Each panel was scribed with an "X" in the middle of the panel. The rust creepage from the scribe was evaluated in accordance with ASTM 1654 method A. The field of the panel was evaluated in accordance with ASTM D1654 method B, ASTM D714, and ASTM D610 for blisters and surface rust. A panel was pulled from testing once one of the two ratings reached "0."

Tables 5 and 6 show the results for panels exposed to ASTM B117 on cold rolled steel with smooth finish. All of the wash primers preformed similarly when primed with MIL-DTL-53022 and MIL-PFR-23377. All of the wash primers produced passing results. Samples where the wash primers were coated with MIL-DTL-53030, corrosion became a huge issue. The panels were corroding from the scribe and blistering. All systems primed with MIL-DTL-53030 were considered to be failing by 336 h. More testing is necessary to determine the exact reason for these failures.

Table 5. ASTM B117 results, evaluated using ASTM 1654 method A, cold rolled steel, smooth finish.

	336 h	672 h	1008 h
N-022-1	7	6	3
N-O22-2	7	6	3
N-O22-3	7	6	3
SP-O22-1	7	6	5
SP-O22-2	6	5	3
SP-O22-3	7	6	3
328-O22-1	6	4	3
328-O22-2	6	6	3
328-O22-3	6	4	3
N-030-1	3	0	
N-030-2	3	0	
N-030-3	2	1	
SP-030-1	5		
SP-030-2	6		
SP-030-3	4		
328-O30-1	5	4	
328-O30-2	7	4	
328-O30-3	5	5	
N-23377-2	8	5	2
N-23377-3	7	5	4
SP-23377-1	8	5	4
SP-23377-2	8	7	7
SP-23377-3	8	7	5
328-23377-1	8	6	5
328-23377-2	8	6	5
328-23377-3	8	7	5

Table 6. ASTM B117 results, evaluated using ASTM D1654 method B in accordance with ASTM D714, and ASTM D610, cold rolled steel, smooth finish.

	336 h	**672 h**	**1008 h**
N-022-1	10	5	3
N-O22-2	10	6	3
N-O22-3	10	6	2
SP-O22-1	7	4	3
SP-O22-2	6	4	2
SP-O22-3	6	0	0
328-O22-1	10	10	0
328-O22-2	10	5	0
328-O22-3	8	5	0
N-030-1	3	0	
N-030-2	4	2	
N-030-3	7	5	
SP-030-1	0		
SP-030-2	0		
SP-030-3	0		
328-O30-1	3	0	
328-O30-2	9	2	
328-O30-3	4	0	
N-23377-2	6	5	3
N-23377-3	10	10	10
SP-23377-1	8	8	6
SP-23377-2	10	10	8
SP-23377-3	10	8	6
328-23377-1	9	5	5
328-23377-2	10	10	10
328-23377-3	10	10	7

Tables 7 and 8 show the results of samples exposed to ASTM B117 on 2024-T3 aluminum with a smooth finish. There is no testing requirement for MIL-DTL-53022 type II for this test. What can be seen is that all three wash primers perform similarly on the smooth aluminum. One major issue is the blistering on the samples treated with control wash primer and primed with MIL-DTL-53030 type I, as seen in table 8. These samples blistered very badly right away. This can be linked to the DOD-P-15328 being sprayed without the thinner, isopropyl alcohol which lead to an inconstant surface and allowed for blistering in the field of the panel. Another issue observed with these panels was that all MIL-DTL-53022 samples completely blistered at the 1008-hour mark. There is no specification test requirement for the MIL-DTL-53022 primer on aluminum at this point. More research would have to be completed to find out if this is an isolated issue with this particular vendor's product or if this is an issue with all MIL-DTL-53022 type II materials

Table 7. ASTM B117 results, evaluated using ASTM 1654 method A 2024-T3 aluminum, smooth finish.

	336 h	**672 h**	**1008 h**	**1344 h**	**1680 h**	**2016 h**	**2352 h**	**2688 h**	**3024 h**
328-022-1	10	10	10						
328-022-2	10	10	10						
328-022-3	9	9	9						
N-022-1	10	10	10						
N-022-2	10	10	10						
N-022-3	9	9	9						
SP-022-1	10	9	9						
SP-022-2	10	10	9						
SP-022-3	10	10	9						
328-030-1	10	10	10	10	10	9	9	9	9
328-030-2	10	10	9	9	9	9	9	9	9
328-030-3	9	9	9	9	9	9	9	9	9
N-030-1	9	9	9	9	9	9	9	9	9
N-030-2	10	10	9	9	9	9	9	9	9
N-030-3	10	10	9	9	9	9	9	9	8
SP-030-1	10	10	10	9	9	9	9	8	6
SP-030-2	10	10	10	9	9	9	9	9	9
SP-030-3	10	10	10	9	9	9	9	9	8
328-377-1	10	9	9	9	5	4	3	3	3
328-377-2	9	9	9	7	6	4	4	4	4
328-377-3	9	9	9	6	5	5	4	4	4
N-377-1	9	9	8	8	8	8	8	8	8
N-377-2	9	8	8	8	8	8	8	8	7
N-377-3	9	9	8	8	8	8	8	8	8
SP-377-1	9	8	8	8	8	8	8	8	8
SP-377-2	9	8	8	8	8	8	8	8	6
SP-377-3	9	8	8	8	8	8	8	8	8

Table 8. ASTM B117 results, evaluated using ASTM D1654 method B in accordance with ASTM D714 and ASTM D610, 2024-T3 aluminum, smooth finish.

	336 h	672 h	1008 h	1344 h	1680 h	2016 h	2352 h	2688 h	3024 h
328-022-1	10	10	0						
328-022-2	10	10	0						
328-022-3	10	10	0						
N-022-1	10	10	0						
N-022-2	10	10	0						
N-022-3	10	10	0						
SP-022-1	10	10	0						
SP-022-2	10	10	0						
SP-022-3	10	10	0						
328-030-1	10	10	10	10	10	10	10	10	9
328-030-2	10	10	10	10	10	10	10	10	9
328-030-3	10	10	10	10	10	10	9	9	9
N-030-1	10	10	10	10	10	10	9	9	9
N-030-2	10	10	10	10	10	10	9	9	9
N-030-3	10	10	10	10	10	10	9	9	8
SP-030-1	2	2	2	2	2	2	2	2	2
SP-030-2	4	4	4	4	4	4	4	4	4
SP-030-3	4	4	4	4	4	4	4	4	4
328-377-1	10	10	10	10	10	10	10	10	10
328-377-2	10	10	10	10	10	10	10	10	10
328-377-3	10	10	10	10	10	10	10	10	10
N-377-1	10	10	10	10	10	10	10	10	10
N-377-2	10	10	10	10	10	10	10	10	10
N-377-3	10	10	10	10	10	10	10	10	10
SP-377-1	10	10	10	10	10	10	10	10	10
SP-377-2	10	10	10	10	10	10	10	10	10
SP-377-3	10	10	10	10	10	10	10	10	10

Tables 9 and 10 displays the results of the test coupons exposed to ASTM B117 on steel with a blast profile. NCP and Spectrum's wash primer performed, as well as the standard wash primer for these coupons. Due to the blast profile, variations will be greater from panel to panel. Overall performance was as expected from each system.

Table 9. ASTM B117 results, evaluated using ASTM 1654 method A, cold rolled steel, blast cleaned.

	336 h	**672 h**	**1008 h**
N-022-1	7	6	4
N-022-2	7	7	4
N-022-3	6	4	0
SP-022-1	5	2	2
SP-022-2	5	3	0
SP-022-3	3	3	3
328-022-1	5	2	1
328-022-2	4	2	2
328-022-3	4	2	2
N-030-1	7	7	7
N-030-2	8	3	2
N-030-3	7	2	1
SP-030-1	5	4	3
SP-030-2	8	5	4
SP-030-3	7	4	4
328-030-1	7	5	5
328-030-2	7	7	6
328-030-3	7	6	5
N-377-1	6	5	4
N-377-2	6	5	5
N-377-3	8	8	7
SP-377-1	7	5	5
SP-377-2	8	5	5
SP-377-3	8	7	6
328-377-1	8	8	7
328-377-2	8	7	6
328-377-3	8	7	7

Table 10. ASTM B117 results, evaluated using ASTM D1654 method B in accordance with ASTM D714 and ASTM D610, cold rolled steel, blast cleaned.

	336 h	672 h	1008 h
N-022-1	8	8	7
N-022-2	8	8	7
N-022-3	8	7	7
SP-022-1	2	2	2
SP-022-2	2	0	0
SP-022-3	4	3	2
328-022-1	4	2	1
328-022-2	7	5	5
328-022-3	5	4	4
N-030-1	8	7	7
N-030-2	10	10	10
N-030-3	6	6	6
SP-030-1	7	6	6
SP-030-2	9	6	6
SP-030-3	6	6	6
328-030-1	6	6	5
328-030-2	10	10	10
328-030-3	10	10	10
N-377-1	10	10	10
N-377-2	10	10	10
N-377-3	10	10	10
SP-377-1	7	7	6
SP-377-2	10	10	10
SP-377-3	10	10	10
328-377-1	10	10	10
328-377-2	10	10	10
328-377-3	10	10	10

Tables 11 and 12 display the results of the test samples exposed to ASTM B117 on aluminum with a blast profile. Again, with the blast profile, greater variations in the results can be expected due to the inconstant surface. This can be seen on table 12 with a few panels that began to blister. The total film thickness was not high enough to completely cover some of the peaks from the blast. This caused blistering that may not have occurred otherwise. Two panels were removed early because of this even though the performance at the scribe was still good. Overall the performances of the experimental wash primers were on par with the control.

Table 11. ASTM B117 results, evaluated using ASTM 1654 method A, 2024-T3 aluminum, blast-cleaned.

	336 h	**672 h**	**1008 h**	**1344 h**	**1680 h**	**2016 h**
328-022-1	10	9	9	9	8	7
328-022-2	10	9	7	6	5	5
328-022-3	10	9	8	7	7	7
N-022-1	10	9				
N-022-2	10	9	8	8	7	6
N-022-3	10	9	9			
SP-022-1	10	9	8	8	7	6
SP-022-2	10	9	9	9	7	7
SP-022-3	10	9	8	8	7	7
328-030-1	10	10	10	10	10	9
328-030-2	10	10	10	10	9	9
328-030-3	10	10	10	9	9	9
N-030-1	10	10	8	8	8	8
N-030-2	10	9	9	8	8	8
N-030-3	10	9	9	9	9	9
SP-030-1	10	9	9	9	9	9
SP-030-2	10	10	10	10	10	9
SP-030-3	10	10	9	9	9	9
328-377-1	10	10	10	9	9	9
328-377-2	10	10	10	9	9	9
328-377-3	10	10	10	9	8	7
N-377-1	10	10	10	9	9	9
N-377-2	10	10	9	9	9	9
N-377-3	10	10	10	9	9	9
SP-377-1	10	10	8	8	8	8
SP-377-2	10	10	9	9	8	8
SP-377-3	10	10	9	9	9	8

Table 12. ASTM B117 results, evaluated using ASTM D1654 method B in accordance with ASTM D714 and ASTM D610, 2024-T3 aluminum, blast cleaned.

	336 h	672 h	1008 h	1344 h	1680 h	2016 h
328-022-1	10	10	10	10	10	10
328-022-2	10	10	10	10	10	7
328-022-3	10	10	10	10	10	10
N-022-1	10	0				
N-022-2	10	10	10	10	10	10
N-022-3	10	3	0			
SP-022-1	10	10	10	10	10	10
SP-022-2	10	9	9	9	10	8
SP-022-3	10	10	10	10	10	10
328-030-1	10	10	10	10	10	6
328-030-2	10	10	10	8	8	8
328-030-3	10	10	10	8	8	8
N-030-1	10	10	8	8	8	8
N-030-2	10	7	7	6	6	6
N-030-3	10	9	8	7	7	7
SP-030-1	10	9	9	8	8	8
SP-030-2	10	10	10	8	7	7
SP-030-3	10	10	8	8	8	8
328-377-1	10	10	10	10	10	10
328-377-2	10	10	10	10	10	10
328-377-3	10	10	10	10	10	10
N-377-1	10	10	10	10	10	10
N-377-2	10	10	10	10	10	10
N-377-3	10	10	10	10	10	10
SP-377-1	10	10	10	10	10	10
SP-377-2	10	10	10	10	10	10
SP-377-3	10	10	10	10	10	10

Tables 13 and 14 show results for GMW 14872 testing on cold rolled steel with a smooth finish. All three wash primers have comparable results. What stands out is the performance of the standard DOD-P-15328 primed with MIL-DTL-53030 type I. These samples blistered very quickly, as seen in table 14. This issue is being investigated to better understand the failure. From what could be determined, free acids from the wash primer surface may have interfered with the cure of the water reducible primer. Wash primers are over indexed with phosphoric acid to etch the substrate. This leaves free acid in the film, thus affecting the water reducible base epoxy. The area exposed to possible free acids from the wash primer would not protect the substrate adequately, causing a failure in the coating system, which will present as blisters.

Table 13. GMW 14872 results, evaluated using ASTM 1654 method A, cold rolled steel, smooth finish.

	10 Cycle	20 Cycle	40 Cycle	60 Cycle	80 Cycle
N-022-1	9	7	7	5	5
N-022-2	7	6	5	5	5
N-022-3	7	7	6	5	5
S-O22-1	8	6	6	5	4
SP-O22-2	7	6	6	5	4
SP-O22-3	8	7	6	5	4
328-O22-1	8	7	6	6	6
328-O22-2	8	7	6	6	5
328-O22-3	9	8	7	7	6
N-030-1	9	6	6	5	4
N-030-2	9	6	5	5	4
N-030-3	9	6	5	4	3
SP-030-1	8	7	7	5	5
SP-030- 2	7	6	3	3	3
SP-030-3	7	6	5	5	4
328-O30-1	9	8	7	7	5
328-O30-2	9	8	7	7	6
328-O30-3	9	8	7	7	7
N-23377-1	6	6	5	5	4
N-23377- 2	8	6	6	5	5
N-23377-3	9	6	5	5	4
SP-23377-1	7	6	6	5	5
SP-23377-2	7	6	6	6	5
SP-23377-3	8	8	7	6	5
328-23377-1	9	9	8	7	7
328-23377-2	9	8	6	6	5
328-23377-3	9	8	7	7	7

Table 14. GMW 14872 results, evaluated using ASTM D1654 method B in accordance with ASTM D714 and ASTM D610, cold rolled steel, smooth finish.

	10 Cycle	20 Cycle	40 Cycle	60 Cycle	80 Cycle
N-022-1	10	10	10	10	10
N-022-2	10	10	10	10	10
N-022-3	10	10	10	10	10
SP-O22-1	10	10	10	10	10
SP-O22-2	10	10	10	10	10
SP-O22-3	10	10	10	10	10
328-O22-1	10	10	10	10	10
328-O22-2	10	10	10	10	10
328-O22-3	10	10	10	10	10
N-030-1	9	9	9	9	9
N-030-2	10	10	10	10	10
N-030-3	10	10	10	10	10
SP-030-1	5	4	3	2	0
SP-030-2	5	5	4	3	3
SP-030-3	7	7	7	7	6
328-O30-1	10	10	7	7	6
328-O30-2	7	6	6	4	4
328-O30-3	4	4	4	4	4
N-23377-1	10	10	10	10	10
N-23377-2	10	10	10	10	10
N-23377-3	10	10	10	10	10
SP-23377-1	10	10	10	10	10
SP-23377-2	10	10	10	10	10
SP-23377-3	10	10	10	10	10
328-23377-1	10	10	10	10	10
328-23377-2	10	10	10	10	10
328-23377-3	10	10	10	10	8

Tables 15 and 16 show results for GMW 14872 testing on 2024-T3 aluminum with a smooth finish. Panels coated with the Spectrum's wash primer and primed with MIL-DTL-53030 type I blistered very quickly. This was the only set that blistered on this substrate in the GMW 14872. All other system had passing results up to 140 cycles.

Table 15. GMW 14872 results, evaluated using ASTM 1654 method A, 2024-T3 aluminum, smooth finish.

	10 Cycle	20 Cycle	40 Cycle	60 Cycle	80 Cycle	100 Cycle	120 Cycle	140 Cycle	160 Cycle
328-022-1	10	9	9	9	9	9	9	8	6
328-022-2	10	9	9	9	9	9	8	7	6
328-022-3	9	9	9	9	9	9	9	9	9
N-022-1	9	9	9	9	9	9	9	8	6
N-022-2	9	9	9	9	5	5	4	2	1
N-022-3	9	9	9	9	9	9	9	9	9
SP-022-1	9	9	9	9	9	9	9	9	9
SP-022-2	9	9	9	9	9	9	9	9	5
SP-022-3	9	9	9	9	9	9	9	9	9
328-030-1	8	8	8	8	8	8	8	8	7
328-030-2	8	8	8	8	8	8	8	7	4
328-030-3	8	8	8	8	8	8	8	7	6
N-030-1	9	9	9	9	9	9	9	8	7
N-030-2	9	9	9	9	9	9	9	7	6
N-030-3	9	9	9	9	9	9	9	8	6
SP-030-1	9	9	8	8	8	8	8	8	6
SP-030-2	9	9	9	9	9	9	9	9	7
SP-030-3	9	9	9	9	9	9	9	9	6
328-377-1	9	9	8	8	8	8	8	7	7
328-377-2	9	9	8	8	8	8	8	7	7
328-377-3	9	8	8	8	8	8	8	8	8
N-377-1	8	8	8	8	8	8	8	8	8
N-377-2	9	9	9	9	9	9	9	9	8
N-377-3	8	8	8	8	8	8	8	8	8
SP-377-1	8	8	8	8	8	8	8	8	8
SP-377-2	8	8	8	8	8	8	8	8	8
SP-377-3	8	8	8	8	8	8	8	8	8

Table 16. GMW 14872 results, evaluated using ASTM D1654 method B in accordance with ASTM D714 and ASTM D610, 2024-T3 aluminum, smooth finish.

	10 Cycle	**20 Cycle**	**40 Cycle**	**60 Cycle**	**80 Cycle**	**100 Cycle**	**120 Cycle**	**140 Cycle**	**160 Cycle**
328-022-1	10	10	10	10	10	10	10	10	10
328-022-2	10	10	10	10	10	10	10	10	10
328-022-3	10	10	10	10	10	10	10	10	10
N-022-1	10	10	10	10	10	10	10	10	10
N-022-2	10	10	10	10	10	10	10	10	10
N-022-3	10	10	10	10	10	10	10	10	10
SP-022-1	10	10	10	10	10	10	10	10	10
SP-022-2	10	10	10	10	10	10	10	10	10
SP-022-3	10	10	10	10	10	10	10	10	9
328-030-1	10	10	10	10	10	10	10	10	10
328-030-2	10	10	10	10	10	10	10	10	10
328-030-3	10	10	10	10	10	10	10	10	10
N-030-1	10	10	10	10	10	10	10	10	10
N-030-2	10	10	10	10	10	10	10	10	10
N-030-3	10	10	10	10	10	10	10	10	10
SP-030-1	4	4	4	4	4	4	4	4	4
SP-030-2	4	4	4	4	4	4	4	4	4
SP-030-3	4	4	4	4	4	4	4	4	4
328-377-1	10	10	10	10	10	10	10	10	10
328-377-2	10	10	10	10	10	10	10	10	10
328-377-3	10	10	10	10	10	10	10	10	10
N-377-1	10	10	10	10	10	10	10	10	10
N-377-2	10	10	10	10	10	10	10	10	10
N-377-3	10	10	10	10	10	10	10	10	10
SP-377-1	10	10	10	10	10	10	10	10	10
SP-377-2	10	10	10	10	10	10	10	10	10
SP-377-3	10	10	10	10	10	10	10	10	10

Tables 17 and 18 show results for GMW 14872 testing on blasted steel. Again, the blasted steel surface provides great variation from sample to sample which can cause blistering and can be seen in table 18. Other than the variation in blistering that can be attributed to the inconsistent profile of the substrate, the experimental wash primer performed on par with control.

Table 17. GMW 14872 results evaluated using ASTM 1654 method A, cold rolled steel, blast cleaned.

	20 Cycle	**40 Cycle**	**60 Cycle**
N-022-1	6	3	3
N-022-2	5	2	2
N-022-3	6	3	3
SP-022-1	5	4	2
SP-022-2	5	3	2
SP-022-3	6	3	1
328-022-1	5	4	4
328-022-2	4	3	3
328-022-3	5	4	4
N-030-1	4	2	1
N-030-2	5	2	2
N-030-3	4	1	1
SP-030-1	6	5	5
SP-030-2	7	5	5
SP-030-3	6	5	5
328-030-1	5	4	3
328-030-2	5	4	4
328-030-3	6	4	4
N-377-1	6	1	0
N-377-2	5	3	1
N-377-3	4	3	3
SP-377-1	6	3	3
SP-377-2	5	5	2
SP-377-3	5	3	3
328-377-1	6	4	3
328-377-2	5	4	3
328-377-3	5	3	1

Table 18. GMW 14872 results, evaluated using ASTM D1654 method B in accordance with ASTM D714 and ASTM D610, cold rolled steel, blast cleaned.

	20 Cycle	40 Cycle	60 Cycle
N-022-1	10	10	10
N-022-2	10	10	10
N-022-3	10	10	10
SP-022-1	10	8	8
SP-022-2	7	5	5
SP-022-3	7	5	0
328-022-1	9	9	8
328-022-2	9	9	9
328-022-3	10	10	10
N-030-1	10	10	10
N-030-2	10	10	10
N-030-3	4	4	4
SP-030-1	7	7	7
SP-030-2	10	7	7
SP-030-3	10	10	10
328-030-1	5	4	4
328-030-2	10	10	10
328-030-3	7	7	7
N-377-1	9	9	9
N-377-2	10	10	10
N-377-3	10	10	10
SP-377-1	9	9	9
SP-377-2	10	10	9
SP-377-3	10	10	10
328-377-1	10	10	10
328-377-2	10	10	10
328-377-3	10	8	8

Tables 19 and 20 show results for GMW 14872 testing on 2024-T3 aluminum with a blast finish. The performance of the experimental wash primers in this test was very consistent with results from the control.

Table 19. GMW 14872 results, evaluated using ASTM 1654 method A, 2024-T3 aluminum, blast cleaned.

	20 Cycle	40 Cycle	60 Cycle	80 Cycle	100 Cycle
328-022-1	9	8	8	7	6
328-022-2	8	6	6	6	5
328-022-3	7	6	5	5	5
N-022-1	7	5	4	3	3
N-022-2	7	5	3	3	2
N-022-3	7	5	4	4	3
SP-022-1	8	6	5	5	5
SP-022-2	7	4	4	3	2
SP-022-3	7	5	4	3	3
328-030-1	9	9	7	6	5
328-030-2	9	9	9	7	7
328-030-3	9	9	8	8	8
N-030-1	7	7	6	6	6
N-030-2	9	7	6	6	5
N-030-3	9	8	7	6	6
SP-030-1	10	10	9	9	7
SP-030-2	10	10	9	9	9
SP-030-3	10	9	9	9	9
328-377-1	10	9	7	6	5
328-377-2	10	8	6	5	5
328-377-3	9	9	8	6	6
N-377-1	9	9	9	9	7
N-377-2	10	7	7	7	6
N-377-3	9	9	9	8	8
SP-377-1	9	7	6	5	5
SP-377-2	8	6	6	5	5
SP-377-3	9	5	5	4	4

Table 20. GMW 14872 results, evaluated using ASTM D1654 method B in accordance with ASTM D714 and ASTM D610, 2024-T3 aluminum, blast cleaned.

	20 Cycle	40 Cycle	60 Cycle	80 Cycle	100 Cycle
328-022-1	10	10	10	10	10
328-022-2	10	10	10	10	10
328-022-3	10	10	10	10	10
N-022-1	10	10	10	10	10
N-022-2	10	10	10	10	10
N-022-3	10	10	10	10	10
SP-022-1	10	10	10	10	10
SP-022-2	10	10	10	10	10
SP-022-3	10	10	10	10	10
328-030-1	10	10	10	7	6
328-030-2	10	10	10	10	7
328-030-3	10	10	10	10	10
N-030-1	10	10	10	10	10
N-030-2	10	10	10	9	8
N-030-3	10	10	8	7	7
SP-030-1	10	10	10	8	8
SP-030-2	10	10	10	10	10
SP-030-3	10	10	10	10	10
328-377-1	10	10	10	10	10
328-377-2	10	10	10	10	10
328-377-3	10	10	10	10	10
N-377-1	10	10	10	10	10
N-377-2	10	10	10	10	10
N-377-3	10	10	10	10	10
SP-377-1	10	10	10	10	10
SP-377-2	10	10	10	10	10
SP-377-3	10	10	10	10	10

All of the corrosion testing for B117 and GMW 14872 was performed on flat test panels prepared by Letterkenny Army Depot or supplied by ARL. The wash primer samples were also tested with a dissimilar metal galvanic test plate. A sample of this test plate can be seen in figure 5. An aluminum test panel with 6 holes was coated with each system. Afterwards, two aluminum, two stainless steel, and two zinc plated bolts were attached in the locations shown in figure 4 to the torque values shown in table 21. One panel from each system was exposed in B-117 salt fog for 336 h and one panel was exposed in GMW 14872 cyclic test for 20 cycles. Afterward, these panels were visually examined for surface defects and signs of galvanic corrosion.

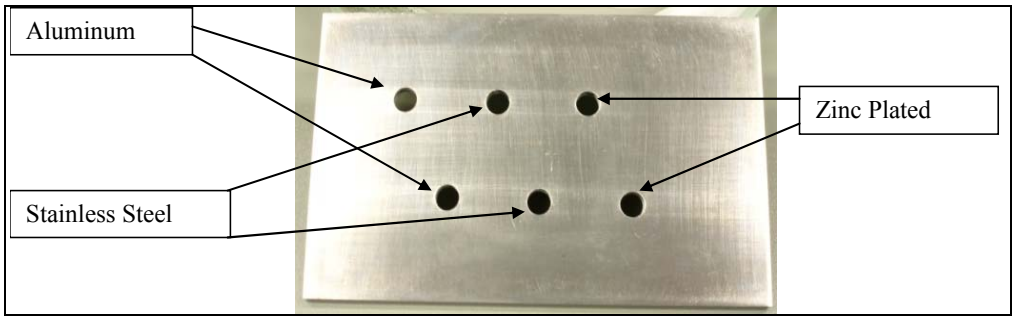

Figure 5. Sample galvanic panel with bolt locations for dissimilar metal testing.

Table 21. Torque values for each bolt material.

Bolt Material	Torque
Aluminum	25 in lb
Zinc Plated	150 in lb
Stainless Steel	79 in lb

The panels were inspected around the area of the washer and under the washer after the exposure. This was the area exposed directly to the elements of each chamber. The backs of each panel were examined but did not receive the direct exposure that the front received in the chambers.

The plates showed no signs of corrosion around the aluminum bolts and the zinc plated bolts with all of the systems. All three wash primers showed no signs of blistering around the bolt hole. Some issues were observed around when examining the stainless steel samples. The standard DOD-P-15328 with MIL-DTL-53030 primer had significant surface blistering around the bolts after the exposure in both the B-117 and the GMW 14872 testing. The blistering was worse on the panel exposed to GMW 14872. The NCP and Spectrum wash primers had signs of small blistering around the stainless steel bolts, although it was not to the degree that the standard DOD-P-15328 had with the MIL-DTL-53030. There were no issues with the stainless steel bolts with all three wash primers when coated with MIL-DTL-53022 or MIL-PRF-23377.

Outdoor weathering is currently ongoing. At this point, it is too early in the exposure to have results. It is expected that results should be coming in in the next 9 to 12 months. The panels will be exposed at the NASA beach corrosion site until failure.

The demonstration was done using 3 storage containers (tricons) which were sprayed at Letterkenny Army Depot March 19−21, 2012. Each tricon was primed with MIL-DTL-53022 Type III primer and topcoated with MIL-DTL-64159 (*12*) type II. Container A, as seen in figure 6, was pretreated with the specification wash primer. Container B, as seen in figure 7, was pretreated with NCP's wash primer. Container C, as seen in figure 8, was pretreated with Spectrum's wash primer. These tricons will sit in the fields at Letterkenny Army depot to weather over the next couple of years. Observations will be taken every couple of months.

Figure 6. Tricon coated with DOD-P-15238 wash primer, MIL-DTL-53022 type III primer and MIL-DTL-64159 type II topcoat in tan 686a.

Figure 7. Tricon coated with NCP's experimental wash primer, MIL-DTL-53022 type III primer and MIL-DTL-64159 type II topcoat in tan 686a.

Figure 8. Tricon coated with Spectrum's experimental wash primer, MIL-DTL-53022 type III primer and MIL-DTL-64159 type II topcoat in tan 686a.

5. Discussion/Path Forward

While outdoor beach corrosion testing is still on-going, results up to this point are promising. As reported by NASA at Cape Canaveral for the first six months of outdoor exposure, corrosion results are all comparable for the two wash primer candidates as compared to the control. From the laboratory testing, it seems that the two experimental samples from NCP and Spectrum could be a temporary solution to the VOC emission issue at Letterkenny Army Depot. Final results from NASA's outdoor corrosion weathering site will be required for final judgment.

Other issues were discovered while testing these products. There are a lot of corrosion and blistering issues with wash primer when primed with water base epoxy primer MIL-DTL-53030. While ARL is looking further into this issue, the evidence of this issue has forced ARL to amend MIL-DTL-53072 to restrict the use of MIL-DTL-53030 over wash primer.

The use of MIL-PRF-23377, class N, produced results that may result in further investigation. Two different qualified products were used and produced, at times, vastly different results. Testing for MIL-PRF-23377 is currently done only on aluminum alloy. More testing would have to be done on ferrous substrates to build ~~a~~ confidence with the material's performance. Further investigation of MIL-PRF-23377, class N, should also include a side-by-side comparison with MIL-DTL-53022 type IV. This product is an enhanced corrosion product that has recently been introduced in the latest revision of that specification. MIL-DTL-53022 type IV should provide improved performance when compared to MIL-DTL-53022 type II that was used in this testing.

Finally, the use of thinner in DOD-P-15328 needs to be addressed. Without the proper mixture for this product, it does not spray properly and will not properly cover the substrate. The gaps in coverage promote unwanted blistering in undamaged areas. The use of thinner will address this by allowing the wash primer to flow properly on the substrate and cover the complete area.

Over the next few months, outdoor data will be collected. Once the outdoor data is received, an informed decision can be made in reference to the path forward. Based upon the six month data from outdoor weathering, a demonstration plan was developed and performed at Letterkenny Army Depot. The demonstration entailed the painting of three Storage containers (Tricon) and will be exposed at Letterkenny for one year with weekly monitoring. Each tricon was pretreated with a different wash primer, the two experimental products and the control, DOD-P-15328, and then primed and topcoated with the same epoxy CARC primer and topcoat. Results from the exposure of the tricons and the outdoor exposure at the NASA test site will provide the information necessary to determine whether these two alternative low VOC wash primers are acceptable products to replace DOD-P-15328 at Letterkenny Army Depot.

Additionally, more lab work will be done to determine the exact reason for the issues witnessed with the use of DOD-P-15328 and MIL-DTL-53030. Until this is determined, MIL-DTL-53072 will continue to restrict the use of MIL-DTL-53030 with DOD-P-15328.

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List of Abbreviations, Symbols, and Acronyms

ARL	U.S. Army Research Laboratory
ASTM	American Society for Testing and Materials
CARC	chemical agent resistant coatings
EPA	Environmental Protection Agency
HAPs	hazardous air pollutants
VOC	volatile organic compound

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